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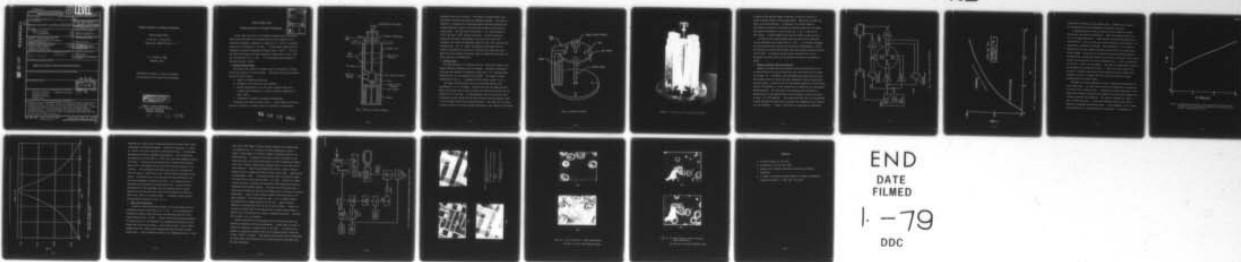
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ACOUSTIC MICROSCOPY AT CRYOGENIC TEMPERATURES.(U)
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ACOUSTIC MICROSCOPY AT CRYOGENIC TEMPERATURES

Annual Summary Report

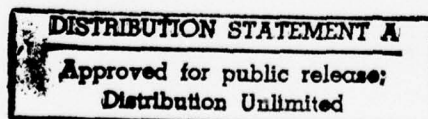
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ANNUAL SUMMARY REPORT

"ACOUSTIC MICROSCOPY AT CRYOGENIC TEMPERATURES"

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In this first year work on the Cryogenic Scanning Acoustic Microscope Project has concentrated on development and testing of components for a reflection instrument to operate at a temperature of 1.95°K in superfluid helium and at a frequency of 500 MHz. At this initial temperature and frequency the acoustic wavelength in helium is about 4500 \AA . To aid in the testing the apparatus has been operated in liquid argon at about 85°K and at frequencies up to 2 GHz. At this frequency the wavelength in the argon is about 4300 \AA .

1. Cryogenic Probe Assembly

The acoustic lens, object and scanning stage are suspended in a pumped helium bath by means of a probe assembly. Objectives that were successfully met for the probe include:

1. Rigid, low vibration construction.
2. Mechanical coarse adjust for lens focussing.
3. Thermal compensation so that lens-object spacing remains near constant during cooldown, i.e. during a change in temperature of about 300°K .
4. Flexibility of design to accommodate modifications.

A diagram of the probe is shown in Fig. 1. Coarse adjust of the focal distance is achieved by a pushrod which is controlled by a differential

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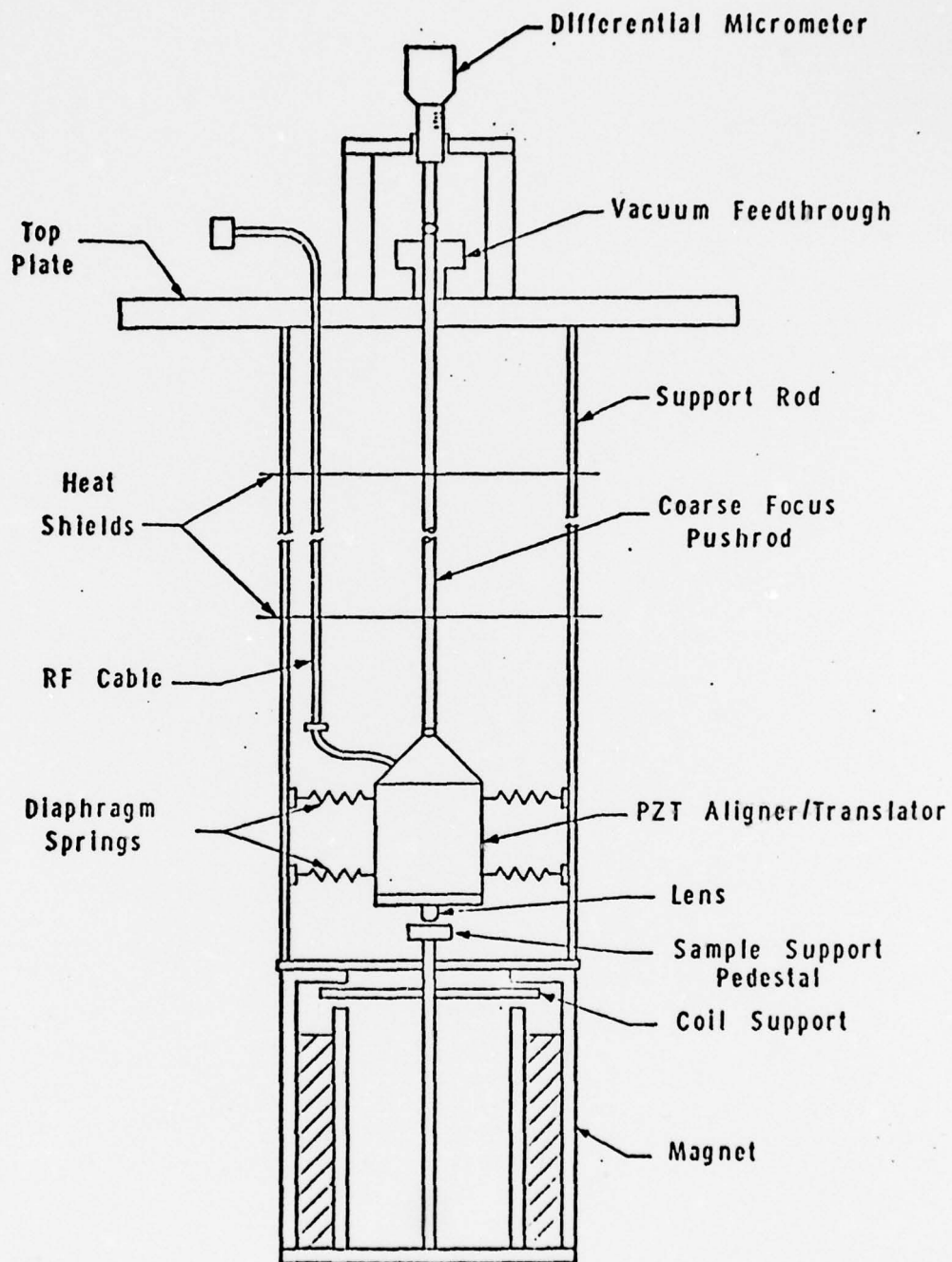


FIG. 1--Diagram of probe assembly.

micrometer head on the top-plate. The pushrod is spring loaded by two diaphragms in the helium and thus has negligible backlash. Fine focus is achieved by a piezoelectric positioning element (Burleigh Instruments PZT Aligner-Translator) located in the helium and acting in series with the coarse adjust. The fine adjust allows about $1 \mu\text{m}$ total movement at 1.95°K and about 100 \AA position resolution. Plenty of electrical connections are made to feedthroughs in the top plate to allow for thermometry and control and monitoring signals for the PZT positioner and scanning stage. The rf signal that generates the acoustic wave is carried out through the top plate on a stainless steel semi-rigid coax. Finally the lens assembly and scanning stage are separate modules which are easily demounted for modification.

2. Scanning Stage

For high resolution to be achieved with the acoustic microscope, a well behaved mechanical scanning system is essential. Therefore, considerable effort has been expended in developing a high quality x-y scanning system compatible with the low temperature environment. The scanner is shown schematically in Fig. 2. A photograph of the scanner appears in Fig. 3.

The sample sits horizontally on top of a flexible aluminum tube approximately 10 cm in length. Attached just below the sample are four small coils of wire that are used to control the motion of the sample and the position of the CRT beam. In each direction, one coil is used for drive by passing a current through the coil which interacts with the magnetic field so as to produce a force in the desired direction. The other coil is used as a sensor to produce an induced voltage proportional to the velocity of the sample.

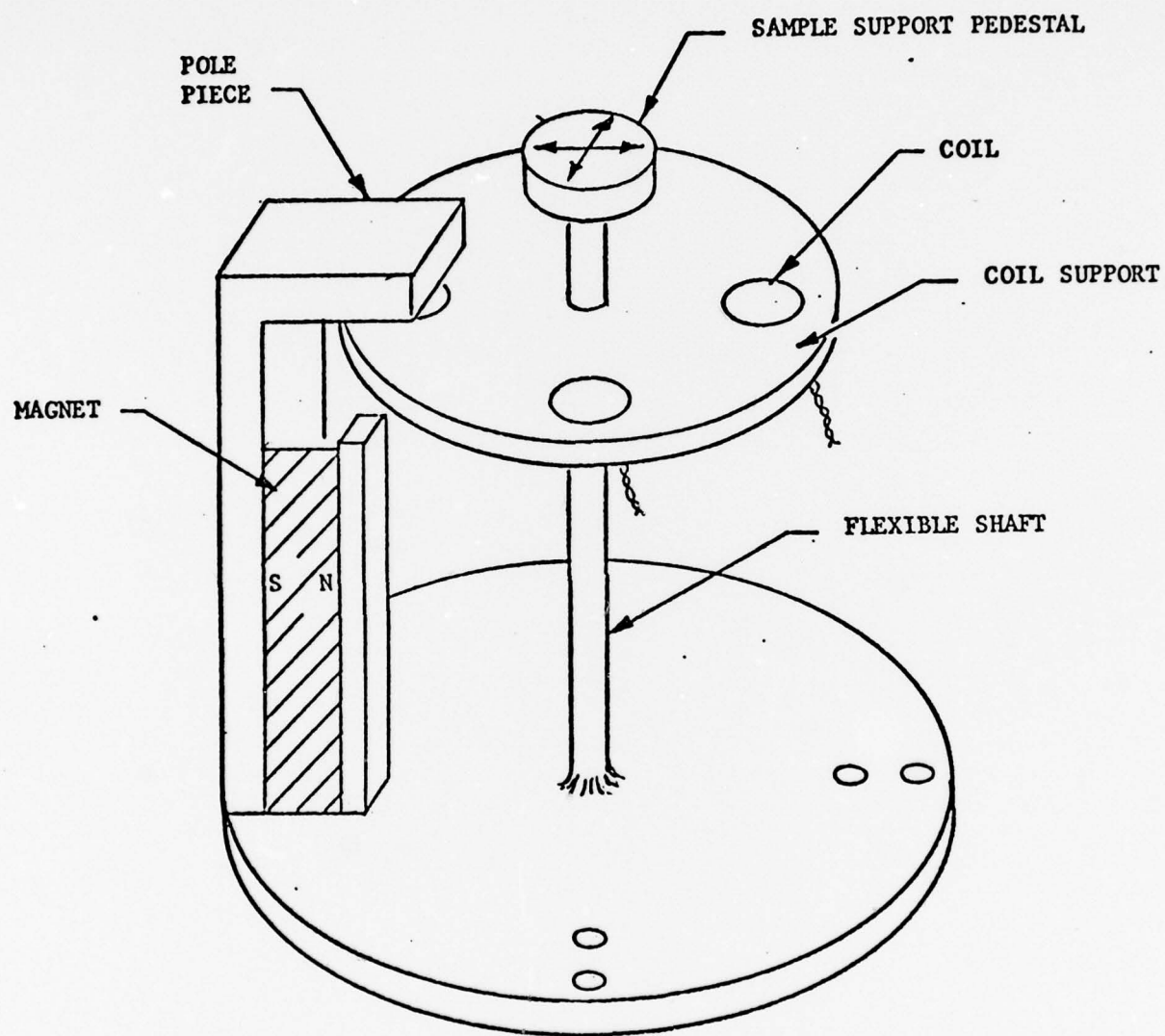


FIG. 2 Diagram of scanner.

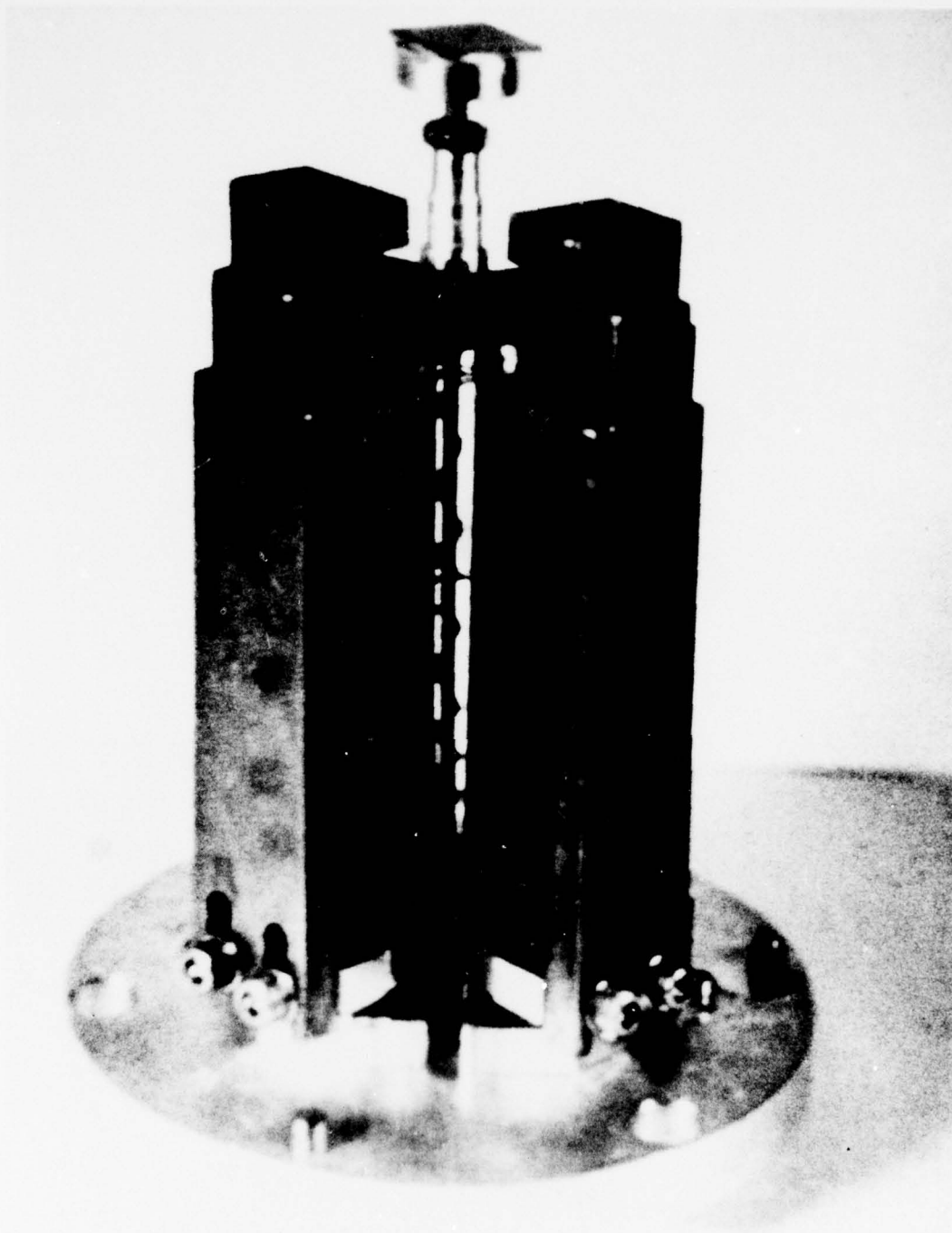


Figure 3 - The cryogenic mechanical scanner

A portion of the velocity signal is fed back to the drive circuitry to provide motional damping of the scanning stage. This serves to reduce the effect of external vibration. In addition, the velocity signal is electronically integrated to determine the relative position of the sample. This position information is used to drive the x and y axes of the CRT display. A block diagram of the scan electronics is shown in Fig. 4.

A version of the scanning system described above has been evaluated at room temperature and in liquid argon at 85°K . As currently configured, the scanning system has a positioning uncertainty of approximately 4000 \AA . This uncertainty arises from electronic noise and externally induced vibration of the sample. An improved scanner has been constructed but not yet evaluated that should reduce the positioning uncertainty to approximately 1500 \AA .

3. Acoustic Transducer and Lens Fabrication

Standard zinc oxide thin film transducers have been fabricated for use in liquid helium on one end of fused quartz rods with either flats or lenses on the other end. In addition, some preliminary experiments have been made with matching layers to improve the power transmission from the fused quartz rods into helium. Because of the very low acoustic impedance of liquid helium ($0.033 \times 10^5 \text{ gm/cm}^2\text{sec}$) a 20 db one-way loss is suffered at the fused quartz-helium interface. The ideal quarter wave matching layer would have an impedance equal to the geometric mean of the impedances of quartz and helium or about $0.7 \times 10^5 \text{ gm/cm}^2\text{sec}$. Few solids exist with such a low longitudinal acoustic impedance but materials are available with impedances in the range of $2-5 \times 10^5 \text{ gm/cm}^2\text{sec}$. Figure 5 illustrates the dependence of transmission

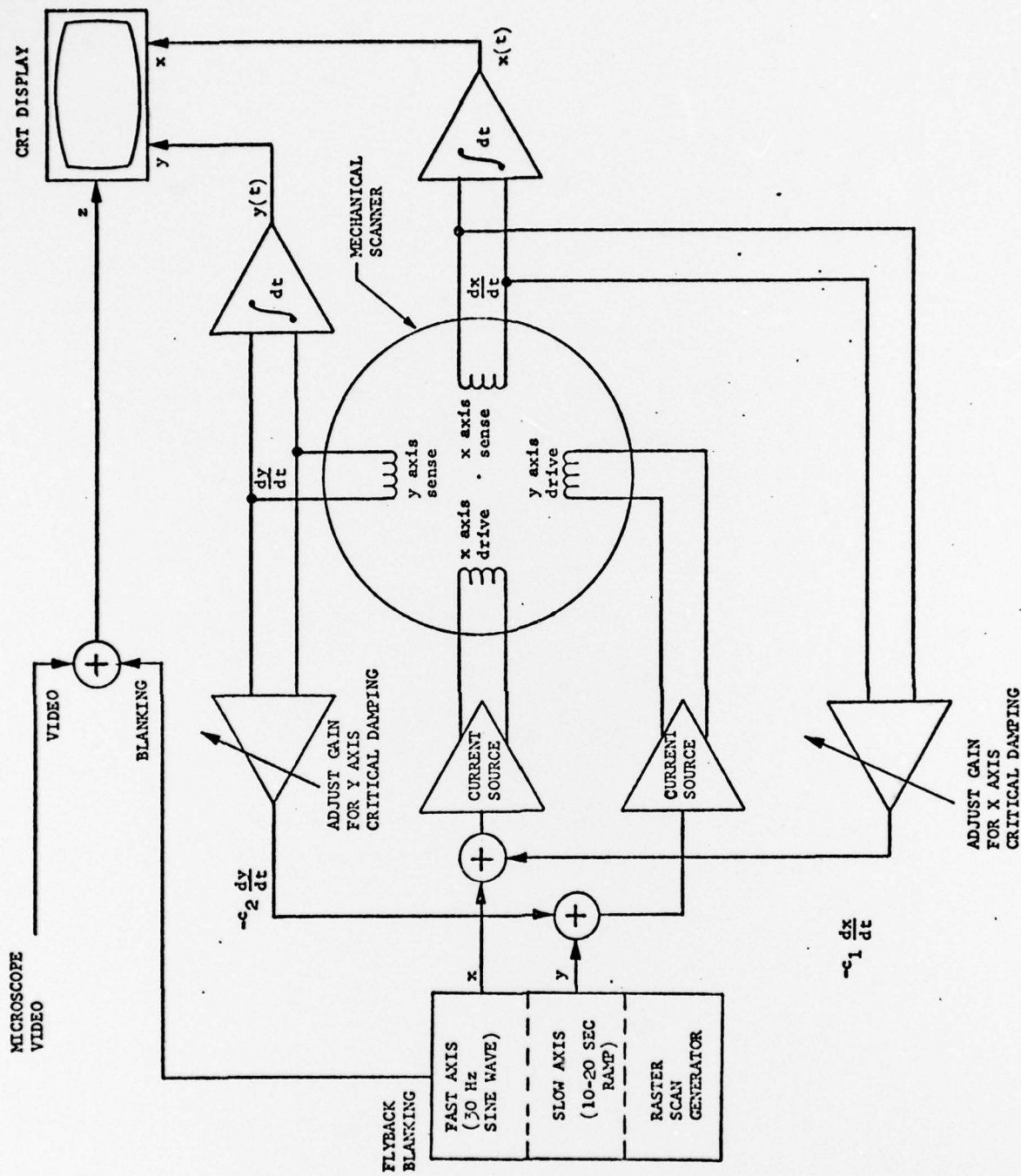


FIG. 4 Block diagram of scan electronics.

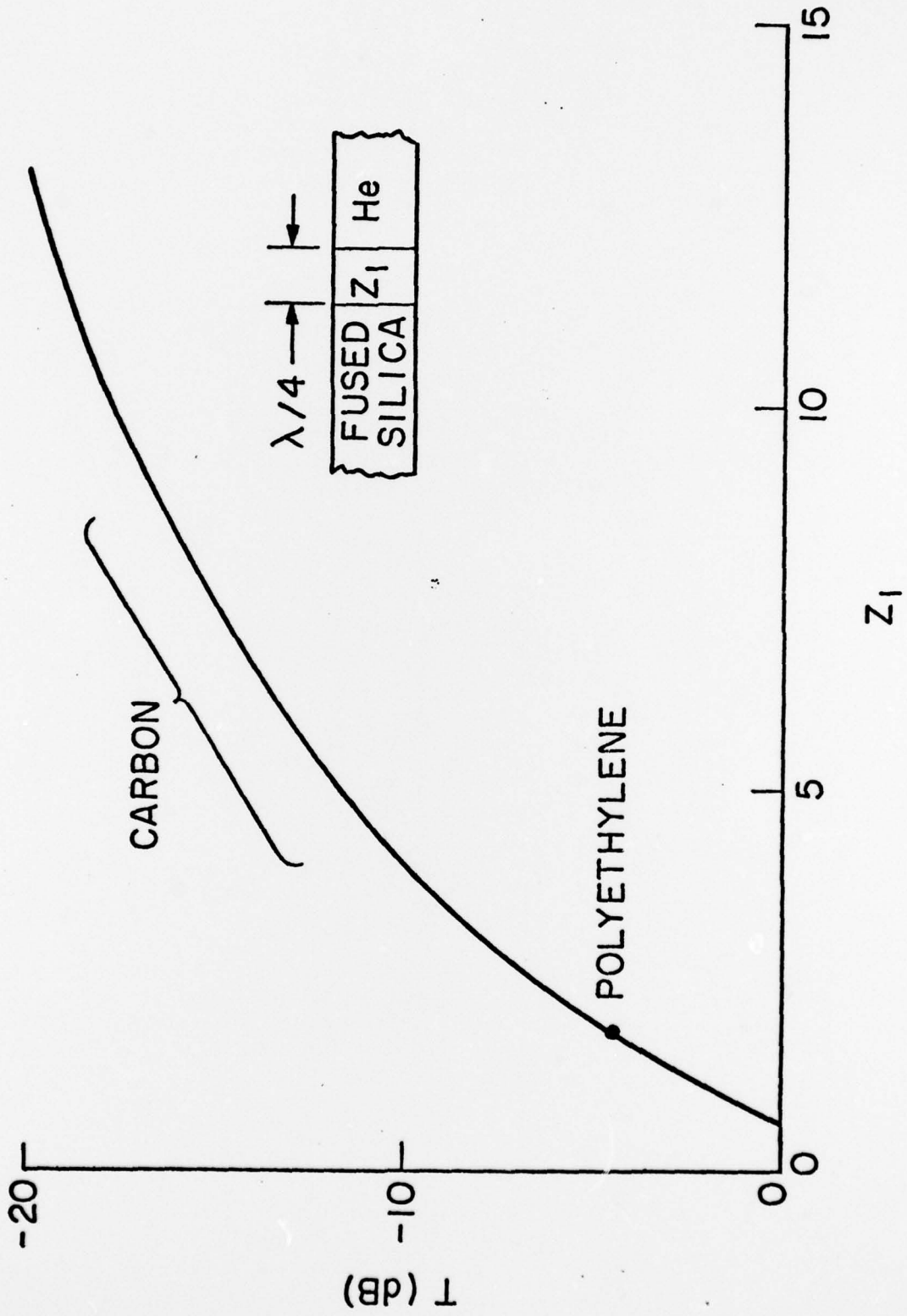


FIG. 5 Transmission coefficient versus impedance of quarter wave matching layer.

coefficient on impedance of the matching layer. Indicated on the figure are two materials we are investigating, polyethylene and carbon.

A surprising amount of work has been done on the problem of coating objects with thin films of polyethylene. White has deposited films up to 10 μm by vacuum deposition.¹ Films can also be cast in thicknesses up to a few microns.² Due to the very low acoustic impedance of polyethylene it is an attractive candidate for matching. We have deposited 1 μm films on fused quartz flats and verified that they can be cycled to low temperatures without damage. We have not yet evaluated the acoustic matching properties of these films. One possible problem is the acoustic attenuation in the plastic. Plastics are notoriously lossy and at frequencies near 1 GHz an attenuation of at least 1 db/ μm can be expected. The effect on transmission coefficient of including loss is indicated in Fig. 6. Since loss in the plastic will increase as frequency squared while layer thickness decreases linearly with frequency eventually a frequency will be reached above which the plastic matching layer is no longer useful.

Although the acoustic impedance of carbon is higher than polyethylene the loss is much lower. For this reason we are also investigating the properties of carbon films as matching layers. Depending on substrate temperature carbon films may be deposited by electron beam deposition or sputtering techniques over a range of densities and hardnesses. The range of impedance for carbon in Fig. 3 reflects this fact. At the lower impedances carbon can be used to fabricate a reasonably good matching layer. We have obtained 1.2 μm carbon films fabricated by e-beam deposition for evaluation.³ We measured the

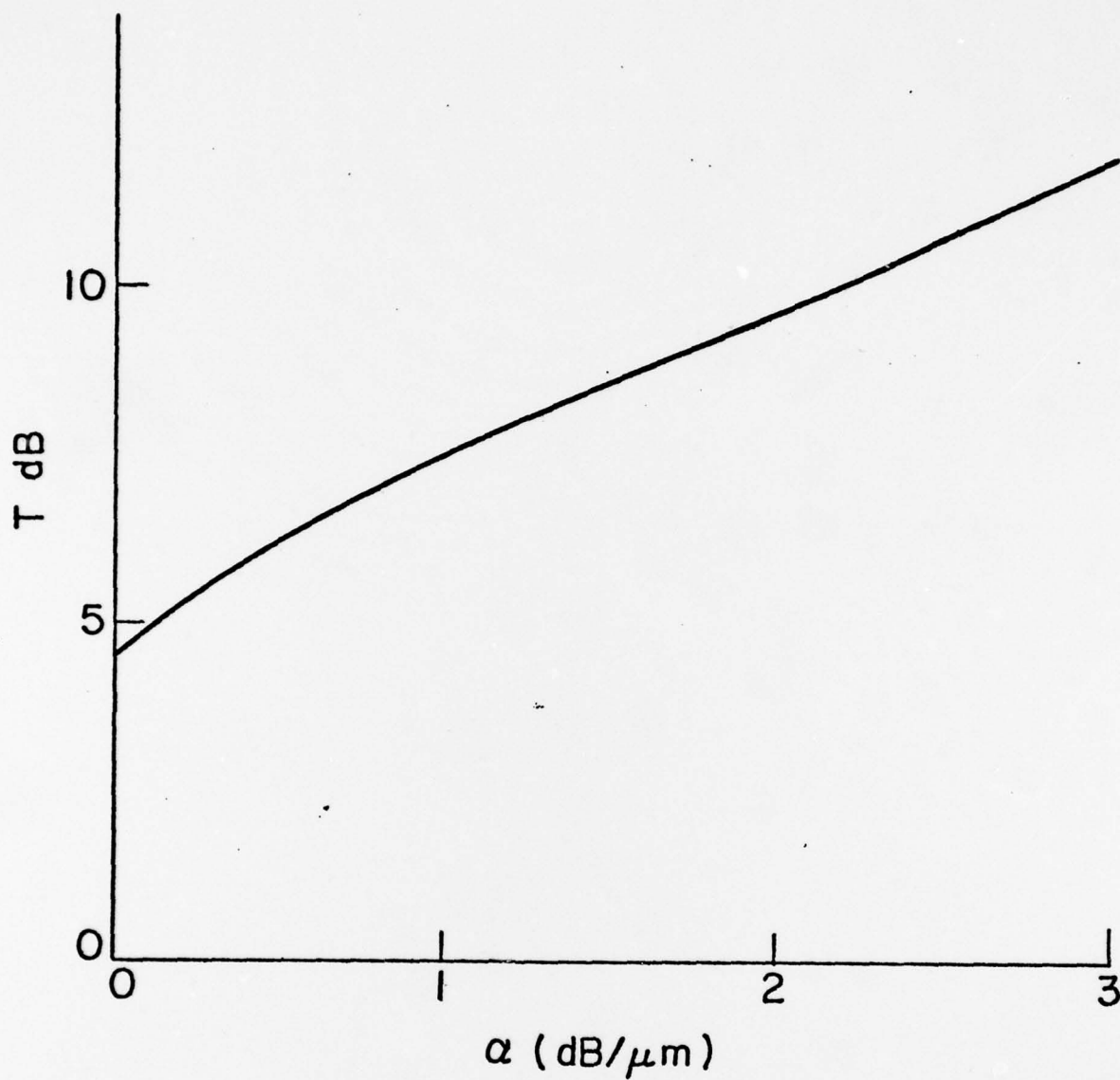


FIG. 6 Transmission coefficient versus attenuation α in quarter wave matching layer for plane waves transmitted from fused silica to helium - see diagram on Fig. 5).

impedance and found it to be near the upper end of the carbon impedance range, however, further attempts are being made under different conditions in hopes of lowering the impedance. We are also beginning a study of sputtered carbon films in our own laboratory to identify which factors specifically lead to low impedance samples.

When a single matching layer of the desired impedance cannot be fabricated it is possible to deposit two films and improve the transmission coefficient at the expense of bandwidth. Examples of some double matching layers and the transmission coefficient expected are indicated in Table I. The calculated frequency response for an Au/Al matching section is shown in Fig. 7. We have experimented with Au/Al and Au/SiO₂ double matching sections in water and found both to be useful, although the aluminum suffered deterioration as a function of time. At low frequencies both combinations were unreliable due to layer separation. At higher frequencies and hence thinner layers this was not a problem.

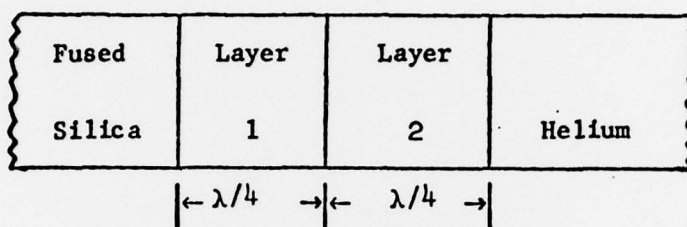
Based on these findings we expect that either plastic or carbon will be used to provide a match at low frequencies during our initial work in helium. As we go to higher frequencies, double matching sections will be used to improve the transmission coefficient still further.

4. Tests of the Complete Assembly using Water and Helium as Propagating Media

Since the apparatus was designed to be used in either water or helium with nearly the same mechanical settings it was possible to conduct initial tests using water. The apparatus could then be cooled to helium temperatures and a focus obtained with a minimum of adjustments. The apparatus was first fitted with a fused quartz rod with no lens (a "flat") and plane waves were

TABLE I

Quarter Wave Matching Sections for Fused Silica to Helium Match



Layer 1	Layer 2	Transmission coefficient (dB)	Comments
None	None	- 20	
Au	Al	- 9	Deteriorates at room temperature especially in presence of H ₂ O
Au	SiO ₂	- 6	good behavior above ~ 700 MHz
Au	Mg	- 5	
W	Mg	- 2	

SI/AU/ AL /HE

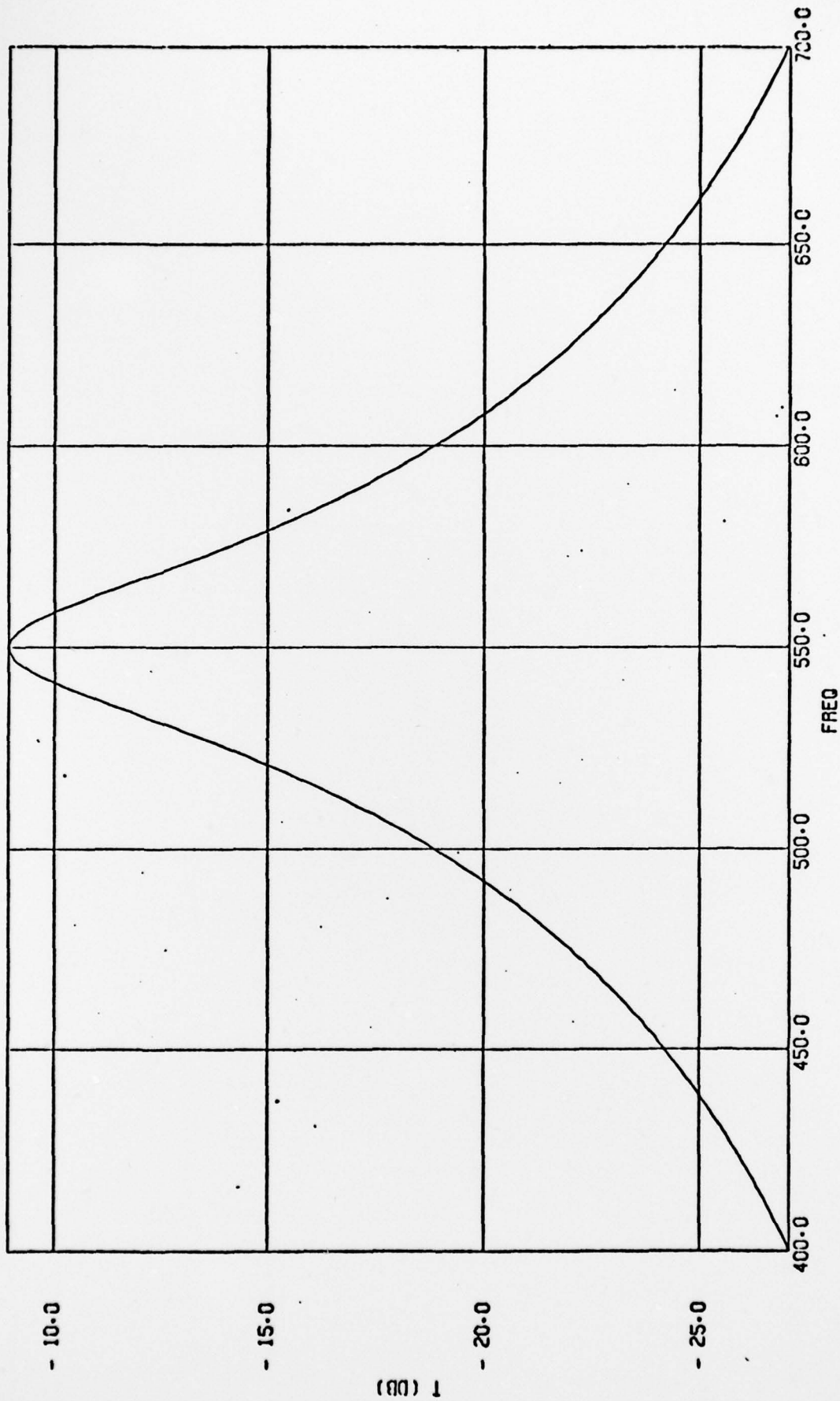


FIG. 7 Calculated frequency dependence of transmission coefficient from fused quartz to helium with Au/Al matching layer.

generated and reflected from a reflecting surface of polished fused silica using water as a propagation medium. Thirty-five nanosecond rf pulses at 500 MHz were used to resolve the reflections in time. The focussing mechanism was tested for smoothness and stability. Next the apparatus was immersed in a helium bath at 1.95°K and a pulse was propagated through the helium. A return pulse was observed but was smaller than expected. This is due to misalignment of the fused silica flat with respect to the reflector. For a plane beam parallelism over the area of the beam must be of the order of 1000 \AA due to the 4500 \AA acoustic wavelength in the helium. During this test it was also noted that the sensitivity of the zinc oxide transducer decreased by less than 1 db during cooldown as determined by reflections in the fused quartz flat. Using this plane wave apparatus it was established that the focussing controls operate smoothly and that the fused quartz flat to object spacing was stable to better than 1000 \AA for several minutes. In addition, during cooldown the same spacing increased by only 10 \mu m .

5. Tests using Liquid Argon

In order to test the microscope further we have used liquid argon as a propagating medium at frequencies up to 2 GHz. At this frequency the wavelength in argon is about the same as the wavelength expected in the initial helium test at 500 MHz. Because of the relatively high impedance of liquid argon ($1.2 \times 10^5 \text{ gm/cm}^2\text{sec}$) the transmission coefficient is much larger than in the case of helium. In our tests we used a 30 \mu m radius sapphire lens with a single glass matching layer which provides a nearly perfect match. Thirty nanosecond pulses at rf frequencies of up to 2 GHz

were used to form images of several objects immersed in the liquid argon. The elements of the rf circuitry are shown schematically in Fig. 8. Figure 9 shows images of a silicon on sapphire integrated circuit at two focal positions. As pointed out by Atalar, contrast and detail in the reflection mode of the acoustic microscope can often be enhanced by imaging with a lens to object spacing either slightly greater or less than the in-focus position.⁴ This shows up dramatically in Fig. 9 where a contrast reversal occurs in changing the focal position a few microns. Magnification is approximately 1600X. The aluminum strips which run across the image from upper left to lower right are about $8\text{ }\mu\text{m}$ wide. We have also imaged a blood cell smear to evaluate the usefulness of the cryogenic microscope in examining fixed biological samples. The blood cells were smeared onto a fused silica substrate and then fixed in methanol prior to cooling to cryogenic temperatures. Figure 10 shows acoustic images of red blood cells at two focal positions. The cells, which are about $7\text{ }\mu\text{m}$ in diameter exhibit the characteristic doughnut shape of the red cell. Again a dramatic difference in contrast and detail is seen in the two images. Figure 11 is an image of another field of the sample in Fig. 10 which contains what we believe to be a white blood cell, perhaps a segmented neutrophil. The white cell is about $12\text{ }\mu\text{m}$ in diameter.

Based on these tests we are confident that the microscope system will function as required in the helium environment. We hope soon to be able to operate the instrument in liquid helium at 500 MHz. It should then be possible to refine the instrument and go up in frequency until a wavelength of about $2000\text{ }\overset{\circ}{\text{A}}$ is reached. This figure is much shorter than the wavelength of visible light and should allow us to resolve objects not observable with the light microscope.

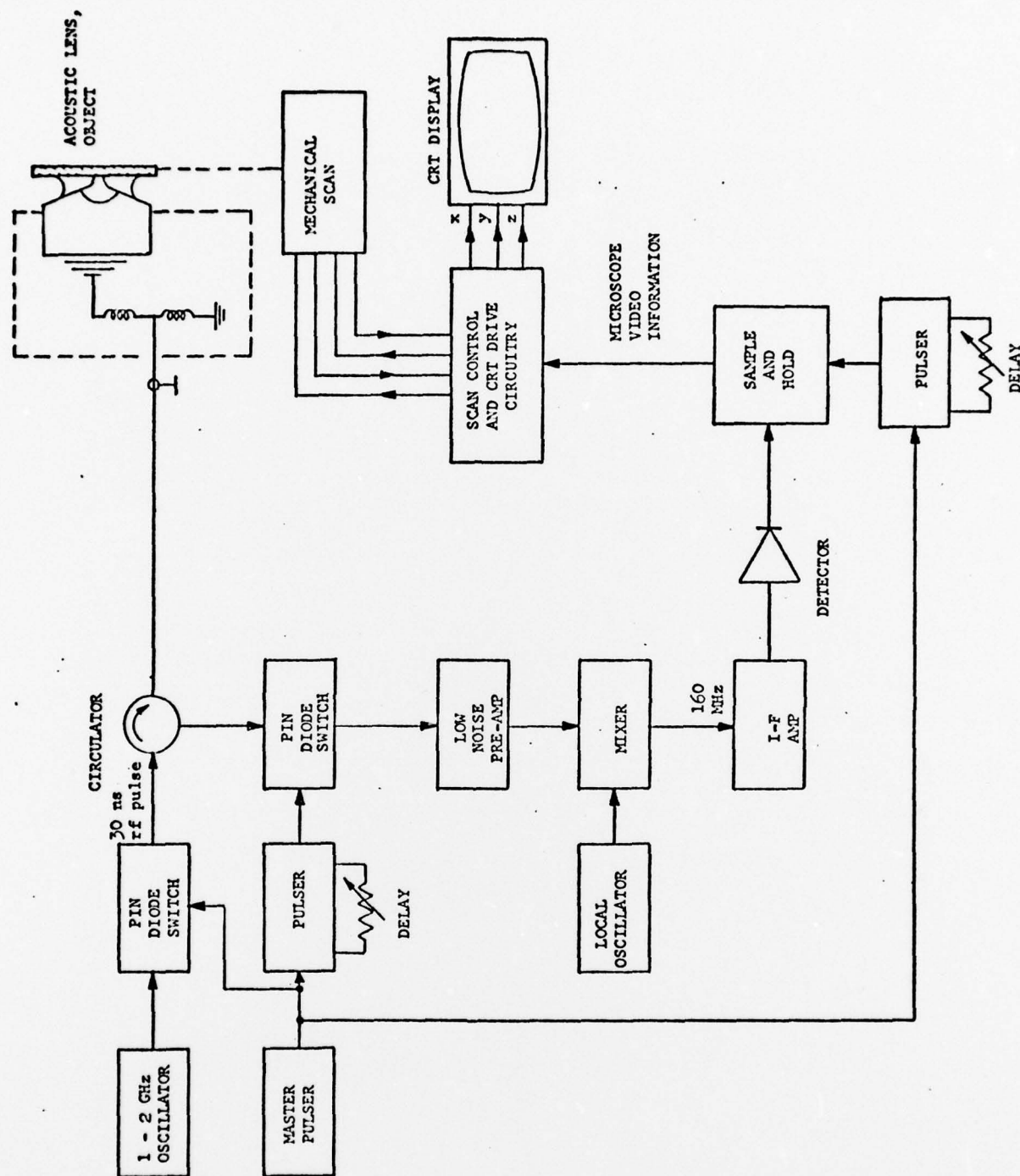
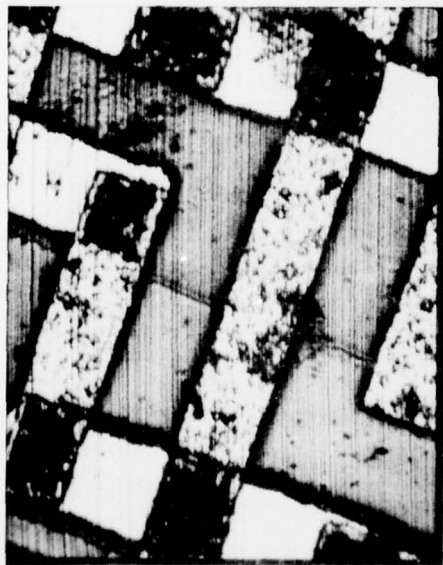


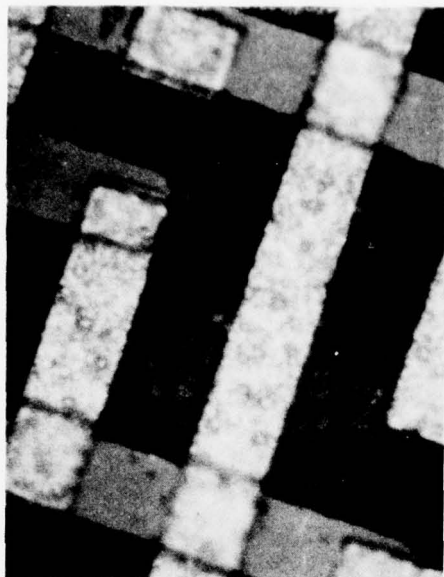
FIG. 8 Block diagram of acoustic microscope using liquid argon.



(a)



(b)



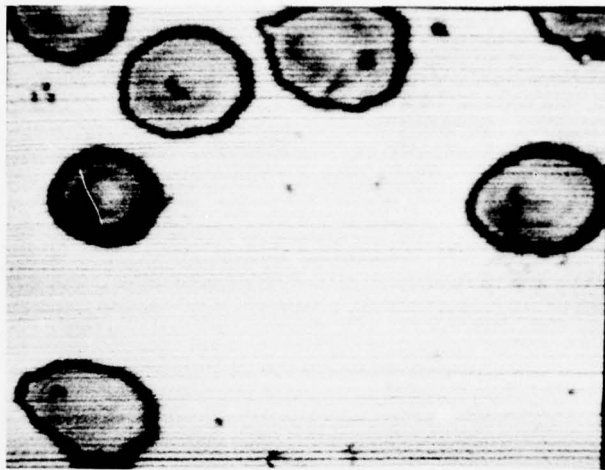
(c)

FIG. 9 Silicon-on-Sapphire Integrated Circuit.
Magnification is 1600X.

(a) Cryogenic acoustic micrograph.

(b) Same as (a) but with different focus.

(c) Optical photo of similar region on different chip.



(a)



(b)

FIG. 10 (a) Red blood cells. 2000X magnification.
(b) Same as (a) but with different focus.



(a)



(b)

FIG. 11 (a) White blood cell (left of center).
1300X magnification.

(b) Same as (a) but with different focus.

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